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Quantitative electron probe microanalysis of lithium in different materials including battery compounds

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Electron probe microanalysis (EPMA) is a reliable and widely used technique to do non-destructive, accurate material characterisation for scientific and industrial applications. Nevertheless, despite of the great interest in lithium (Li) and the urgent need to do accurate non-destructive analysis at a micron scale, quantification of Li using EPMA has not been performed successfully yet. Recently developed periodic multilayers allow spectroscopy in the energy range around the characteristic Li K emission ~ 50 eV [1] but the detection and quantification of Li by a microprobe equipped with a bent crystal spectrometer and standard commercialised multilayers without diffraction gratings still is challenging.

The difficulties of Li detection are caused by different factors: the fluorescence yield of Li is extremely low, few characteristic photons are produced by the decays of the Li 1s core hole, favoring the emission of Auger electrons. Because of their low energy, the photons are strongly absorbed before even leaving the sample and its eventual coating. The signal therefore is principally coming from a thin surface layer that can be subject to contamination and that may be sensitive to electron bombardment. Further photon absorption by the microprobe components, especially by separation windows, will additionally decrease the measured intensity. As the Li K emission (2p – 1s transition) involves valence electrons, the shape of the Li emission band is highly dependent on the density of states (DOS) of the valence band and so highly dependent on the chemical state of the sample. Chemical shifts of some eV and strong peak shape alterations can occur and should be expected for EPMA of light elements [2], complicating quantitative analysis.

This work shows, to our knowledge for the first time, quantitative EPMA of Li in different materials including battery compounds and metal alloys with Li concentrations down to 2 %. This was made possible after the integration of a new detection system. Further work on the quantification procedure using real standards and correction programs suited for low voltage EPMA has also been accomplished. We show that EPMA is a powerful tool to do quantitative analysis of Li even if it is contained in heavy matrices with elements showing characteristic emission bands in the same spectral range as Li. This novel Li quantification method is more accessible than other techniques using synchrotron radiation and less expensive than detection using a SEM equipped with multilayer gratings.

References

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- [2] Schweizer, P., Brackx, E., Jonnard, P., *X-Ray Spectrom.* 2022, *51* (4), 403. <https://doi.org/10.1002/xrs.3290>